

REPORT DOCUMENTATION PAGE

Form Approved OMB NO. 0704-0188

The public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number.
PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.

1. REPORT DATE (DD-MM-YYYY) 25-01-2016	2. REPORT TYPE Final Report	3. DATES COVERED (From - To) 22-Jul-2013 - 21-Jul-2014		
4. TITLE AND SUBTITLE Final Report: Bioactive Encapsulation for Military Food Applications: Request for Enhanced Nano and Micro Particle Fabrication and Characterization Facilities		5a. CONTRACT NUMBER W911NF-13-1-0257		
		5b. GRANT NUMBER		
		5c. PROGRAM ELEMENT NUMBER 611103		
6. AUTHORS David McClements		5d. PROJECT NUMBER		
		5e. TASK NUMBER		
		5f. WORK UNIT NUMBER		
7. PERFORMING ORGANIZATION NAMES AND ADDRESSES University of Massachusetts - Amherst Research Administration Building 70 Butterfield Terrace Amherst, MA 01003 -9242		8. PERFORMING ORGANIZATION REPORT NUMBER		
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS (ES) U.S. Army Research Office P.O. Box 12211 Research Triangle Park, NC 27709-2211		10. SPONSOR/MONITOR'S ACRONYM(S) ARO		
		11. SPONSOR/MONITOR'S REPORT NUMBER(S) 63544-LS-RIP.1		
12. DISTRIBUTION AVAILABILITY STATEMENT Approved for Public Release; Distribution Unlimited				
13. SUPPLEMENTARY NOTES The views, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy or decision, unless so designated by other documentation.				
14. ABSTRACT The purpose of this project was to establish state-of-the art facilities to develop nanoparticle and microparticle encapsulation systems for bioactive components that may improve soldier performance, e.g., nutrient delivery systems or antimicrobial delivery systems. As part of the project we purchased facilitates to fabricate nanoparticles (spray drying unit) and equipment to characterize the powders produced (freeze drying, sputter coating, and scanning electron microscopy). During the course of the project we have developed a wide range of nanoparticle and microparticle delivery systems that can be used to encapsulate nutrient and release bioactive components.				
15. SUBJECT TERMS Bioactive Encapsulation				
16. SECURITY CLASSIFICATION OF: a. REPORT UU		17. LIMITATION OF ABSTRACT UU	15. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON David McClements
b. ABSTRACT UU				19b. TELEPHONE NUMBER 413-545-1019
18. DISTRIBUTION CODE				

Report Title

Final Report: Bioactive Encapsulation for Military Food Applications: Request for Enhanced Nano and Micro Particle Fabrication and Characterization Facilities

ABSTRACT

The purpose of this project was to establish state-of-the art facilities to develop nanoparticle and microparticle encapsulation systems for bioactive components that may improve soldier performance, e.g., nutrient delivery systems or antimicrobial delivery systems. As part of the project we purchased facilitates to fabricate nanoparticles (spray drying unit) and equipment to characterize the powders produced (freeze drying, sputter coating, and scanning electron microscopy). During the course of the project we have developed a wide range of nanoparticle and microparticle delivery systems that can be used to encapsulate, protect, and release bioactive components, including biopolymer complexes, protein nanoparticles, lipid nanoparticles, and microgels. For example, we have encapsulated natural antimicrobials (such as essential oils and polylysine), vitamins (vitamin A, D, and E), and nutraceuticals (curcumin, betacarotene, w-3 fatty acids, polyphenols, and Coenzyme Q10). Vitamins and nutraceuticals may promote soldier performance, whereas antimicrobials may be used to improve the shelf life and safety of rations.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

Received Paper

TOTAL:

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

Received Paper

TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

Number of Presentations: 0.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

Received Paper

TOTAL:

Number of Manuscripts:

Books

Received Book

TOTAL:

Received Book Chapter

TOTAL:

Patents Submitted

Patents Awarded

Awards

Graduate Students

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
-------------	--------------------------

FTE Equivalent:

Total Number:

Names of Post Doctorates

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
-------------	--------------------------

FTE Equivalent:

Total Number:

Names of Faculty Supported

<u>NAME</u>	<u>PERCENT_SUPPORTED</u>
-------------	--------------------------

FTE Equivalent:
Total Number:

Names of Under Graduate students supported

<u>NAME</u>	<u>PERCENT_SUPPORTED</u>
-------------	--------------------------

FTE Equivalent:
Total Number:

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period:

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:.....

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:.....

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):.....

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:.....

The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense

The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields:.....

Names of Personnel receiving masters degrees

<u>NAME</u>

Total Number:

Names of personnel receiving PHDs

<u>NAME</u>

Total Number:

Names of other research staff

<u>NAME</u>	<u>PERCENT_SUPPORTED</u>
-------------	--------------------------

FTE Equivalent:
Total Number:

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

Equipment was purchased.

Technology Transfer

Bioactive Encapsulation for Military Food Applications: Request for Enhanced Nano- & Micro-particle Fabrication & Characterization Facilities

Introduction

The purpose of this project was to establish state-of-the art facilities to develop nanoparticle and microparticle encapsulation systems for bioactive components that may improve soldier performance, e.g., nutrient delivery systems or antimicrobial delivery systems. As part of the project we purchased facilitates to fabricate nanoparticles (spray drying unit) and equipment to characterize the powders produced (freeze drying, sputter coating, and scanning electron microscopy). During the course of the project we have developed a wide range of nanoparticle and microparticle delivery systems that can be used to encapsulate, protect, and release bioactive components, including biopolymer complexes, protein nanoparticles, lipid nanoparticles, and microgels. For example, we have encapsulated natural antimicrobials (such as essential oils and polylysine), vitamins (vitamin A, D, and E), and nutraceuticals (curcumin, beta-carotene, w-3 fatty acids, polyphenols, and Coenzyme Q10). Vitamins and nutraceuticals may promote soldier performance, whereas antimicrobials may be used to improve the shelf life and safety of rations.

The physicochemical properties of nanoparticle and microparticle encapsulation systems have been characterized, including particle size distribution, electrical charge, and morphology. In addition, the biological activity of antimicrobials has been established using model microorganisms, whereas the potential bioavailability of nutraceuticals has been established using a simulated gastrointestinal tract (GIT) model that includes mouth, stomach, and small intestine phases.

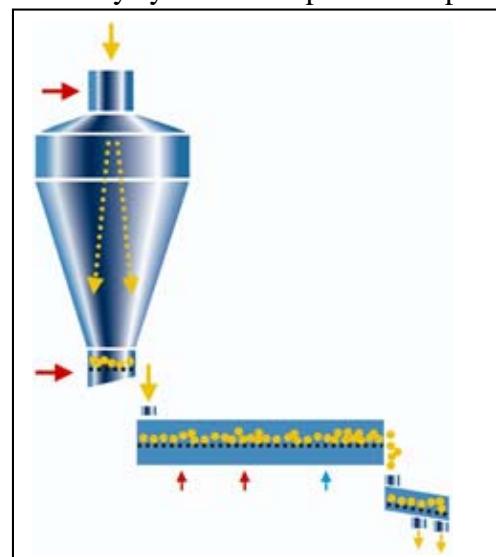


Figure 1: Schmatic diagram of the principles of a small scale spray drier that can be used to covert fluid colloidal delivery systems into agglomerated powders (from Niro.com).

Particle Fabrication Facilities

Spray drier – A small-scale spray drier was purchased to convert fluid delivery systems into solid powders using *microencapsulation*. Microencapsulation involves passing a fluid through a tiny nozzle to form a mist of small drops, which are then dried by evaporating the solvent at elevated temperatures (**Figure 1**). This unit has been used for drying delivery systems. The instrument purchased mimics the drying conditions that occur within industrial scale spray driers, so as to facilitate scale-up from pilot plant to production. The advantage of purchasing a small-scale spray drier is that it can be utilized to rapidly screen the efficacy of different construction materials and structural organization principles used in developing delivery systems. This has provided us with the ability to test different wall materials in the powders that can protect the nutraceuticals during storage by inhibiting oxygen and light exposure, while also enhancing the bioaccessibility of the microencapsulated nutraceutical after they are dispersed in foods and beverages.

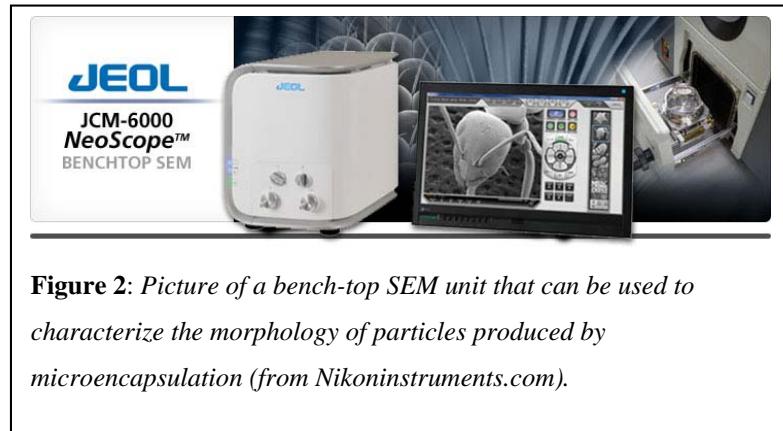


Figure 2: Picture of a bench-top SEM unit that can be used to characterize the morphology of particles produced by microencapsulation (from Nikoninstruments.com).

Particle Characterization Facilities

Bench-top SEM – A bench-top scanning electron microscope (SEM) was purchased to characterize the morphology and dimensions of the structured delivery systems prepared using the microencapsulation method. This instrument enables the microstructure of materials to be determined by passing an electron beam across their surface (**Figure 2**). The functional performance of bioactive components is known to be strongly influenced by their nano- and micro-structure, and so knowledge of their morphology is essential for understanding and controlling their stability and properties.

Applications

The equipment is being used to educate students on the fabrication and characterization of nanoparticle and microparticle delivery systems, as well in fundamental research aimed at designing delivery systems to: improve the stability of bioactive agents in food products/rations; their dispersibility in food products and rations; and enhance their biological activity (such as antimicrobial activity or bioavailability).

Representative Publications

Komaiko, J., & McClements, D. J. (2014). Optimization of isothermal low-energy nanoemulsion formation: Hydrocarbon oil, non-ionic surfactant, and water systems. *Journal of Colloid and Interface Science*, 425, 59-66.

Komaiko, J., & McClements, D. J. (2015c). Low-energy formation of edible nanoemulsions by spontaneous emulsification: Factors influencing particle size. *Journal of Food Engineering*, 146, 122-128.

Landry, K. S., Chang, Y., McClements, D. J., & McLandsborough, L. (2014). Effectiveness of a novel spontaneous carvacrol nanoemulsion against *Salmonella enterica Enteritidis* and *Escherichia coli O157:H7* on, contaminated mung bean and alfalfa seeds. *International Journal of Food Microbiology*, 187, 15-21.

Lopez-Pena, C. L., & McClements, D. J. (2014a). Optimizing delivery systems for cationic biopolymers: Competitive interactions of cationic polylysine with anionic K-carrageenan and pectin. *Food Chemistry*, 153, 9-14.

Lopez-Pena, C. L., & McClements, D. J. (2014c). Optimizing delivery systems for cationic biopolymers: Competitive interactions of cationic polylysine with anionic kappa-carrageenan and pectin. *Abstracts of Papers of the American Chemical Society*, 247.

Ozturk, B., Argin, S., Ozilgen, M., & McClements, D. J. (2014). Formation and stabilization of nanoemulsion-based vitamin E delivery systems using natural surfactants: Quillaja saponin and lecithin. *Journal of Food Engineering*, 142, 57-63.

Saberi, A. H., Fang, Y., & McClements, D. J. (2014a). Effect of Salts on Formation and Stability of Vitamin E-Enriched Mini-emulsions Produced by Spontaneous Emulsification. *Journal of Agricultural and Food Chemistry*, 62 (46), 11246-11253.

Saberi, A. H., Fang, Y., & McClements, D. J. (2014b). Stabilization of vitamin E-enriched mini-emulsions: Influence of organic and aqueous phase compositions. *Colloids and Surfaces a-Physicochemical and Engineering Aspects*, 449, 65-73.

Saberi, A. H., Fang, Y., & McClements, D. J. (2014c). Stabilization of Vitamin E-Enriched Nanoemulsions: Influence of Post-Homogenization Cosurfactant Addition. *Journal of Agricultural and Food Chemistry*, 62 (7), 1625-1633.

Yao, M., Xiao, H., & McClements, D. J. (2014). Delivery of Lipophilic Bioactives: Assembly, Disassembly, and Reassembly of Lipid Nanoparticles. *Annual Review of Food Science and Technology*, Vol 5, 5, 53-81.

Yi, J., Zhu, Z., McClements, D. J., & Decker, E. A. (2014). Influence of Aqueous Phase Emulsifiers on Lipid Oxidation in Water-in-Walnut Oil Emulsions. *Journal of Agricultural and Food Chemistry*, 62 (9), 2104-2111.

Zhang, Z., Decker, E. A., & McClements, D. J. (2014). Encapsulation, protection, and release of polyunsaturated lipids using biopolymer-based hydrogel particles. *Food Research International*, 64, 520-526.

Zheng, J., Li, Y., Song, M., Fang, X., Cao, Y., McClements, D. J., & Xiao, H. (2014). Improving intracellular uptake of 5-demethyltangeretin by food grade nanoemulsions. *Food Research International*, 62, 98-103.